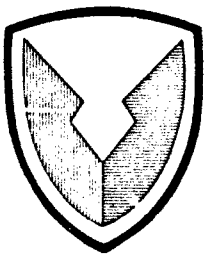
AD

67-2254

DEVELOPMENT OF A WIDE-TEMPERATURE RANGE HYDRAULIC FLUID



TECHNICAL REPORT

By

Ralph L. LeMar

September 1967

U. S. ARMY WEAPONS COMMAND

ROCK ISLAND ARSENAL

RESEARCH & ENGINEERING DIVISION

33

AD

U. S. ARMY WEAPONS COMMAND

ROCK ISLAND ARSENAL

RESEARCH & ENGINEERING DIVISION

TECHNICAL REPORT

67-2254

DEVELOPMENT OF A WIDE-TEMPERATURE RANGE HYDRAULIC FLUID

By

Ralph L. LeMar
Research Laboratories

September 1967

DA # 1C024401A108 AMS Code 5025.11.802

ABSTRACT

Work was performed to formulate an extreme low temperature hydraulic fluid that would show better physical and chemical stability at high temperatures than do the commonly used low temperature mineral oil based fluids.

A synthetic fluid, tetra(2-ethylhexyl) orthosilicate, was selected as a base stock for the fluid. Various chemicals were examined for their effects on the base fluid's thermal stability and other properties. These chemicals included oxidation inhibitors, hydrolysis inhibitors, thickeners, and rubber swell improvers.

Thermal stability tests at 700°F. showed the silicate fluid to be adversely affected by several frequently used additives. Unreactive, aromatic compounds were found that were compatible with the silicate fluid and also improved its rubber swell properties.

A promising blend, No. 7M8, was evolved and evaluated for properties pertinent to hydraulic fluid use. Results indicated that this blend's properties were generally as good as or better than those of standard low temperature mineral oil based fluids.

An outline of requirements for a Military Specification was prepared.

FOREWORD

The work reported here was performed under DA Project No. 1C024401A108, AMS Code 5025.11.802 on Power Transmission Fluids under work unit title, "Determine Significant Physical and Chemical Properties of Experimental High Temperature Fluids." It was carried out for the purposes of: (1) Formulating a stable, wide temperature range fluid and (2) Describing the properties of the fluid.

TABLE OF CONTENTS

PROBLEM

To formulate and describe the properties of a hydraulic fluid that possessed good low temperature properties as well as satisfactory stability for use at high temperatures.

BACKGROUND

Hydraulic systems are expected to perform satisfactorily at wide extremes of temperatures. In the past, low temperature operation at -65°F. or lower, has generally been accomplished by the use of fluids based on a light mineral oil, e.g., those described by Specification MIL-H-46004A.[1] These usually contain small amounts of additives to improve antiwear, oxidation resistance and foam resistance properties. Some specification oils, such as MIL-H-8301[illegible],[2] contain polymeric viscosity index improvers. Two other extensively used fluids described by Military Specification, MIL-H-5606B[3] and the corrosion preventive version, MIL-H-6083C[4] may possess comparatively high viscosities of 3000 to 3500 centistokes at -65°F. All of the above fluid's mineral oil base stocks are volatile and so show low flash and fire points. This inherent drawback has been somewhat corrected by the deep dewaxing of mineral oils. However, a fluid based on such a mineral oil, MIL-H-27601A,[5] is limited to use at temperatures above -40°F., though its properties at high temperatures are superior to those of the light gas oils used in the previously discussed fluids.

Synthetic base stocks must be used to achieve a better balance between low temperature fluidity and high temperature stability. Among the more versatile of these are the silicates and disiloxanes. One of these, hexa(2-ethylbutyl) disiloxane, is the base fluid utilized in MIL-H-8446B.[6] This specification fluid contains a polymeric thickener, an ester for improvement of rubber swell properties and an inhibitor to prevent oxidative and hydrolytic breakdown of the fluid. Because of the thickener, this fluid shows a maximum viscosity of 2,500 centistokes at -65°F., which makes it of marginal use at lower temperatures. Moreover, additives used in the fluids can compromise their high temperature stability.

Another Military Specification fluid that utilizes a silicate base fluid is MIL-L-14107B.[7] It contains a corrosion inhibitor, usually of the sulfonate type. This fluid does show good low temperature capabilities, but its thermal stability is compromised, both by the instability of the inhibitor as well as by probable interactions between the inhibitor and base fluid at high temperatures. Hatton[8] has described several commercial products based on the

silicate fluids. These are said to show good thermal stability, fair lubrication and good viscosity temperature properties. On the negative side, they are subject to oxidative and hydrolytic deterioration and do not swell elastomeric seals and gaskets to the degree needed for the effective function of these components.

Both commercial specialty products and military specification fluids have been adequately inhibited for oxidation and hydrolytic stability. However, the use of additives for improving rubber swell characteristics generally contributed to silicate base fluid instability. For example, Baum[9] found that the di(2-ethylhexyl) sebacate used at a 15% concentration in a disiloxane type MIL-H-8446A fluid was very deleterious to the hydrolytic stability of the base fluid. Similarly, in work at this Research Laboratory,[10] thermal stability tests at 650°F. indicated that a similar disiloxane blend containing a diester showed greater sludge formation, metal attack and viscosity loss after thermal degradation. Peeler and Kovacich[11] evaluated many additives for improving the rubber swell characteristics of silicate fluids. Generally, only ester type fluids were effective, when both rubber swell improvement and low temperature stability were considered.

Silicate fluids, however, had two qualities essential to a wide temperature fluid, i.e., good thermal stability and excellent low temperature properties. One of these that had been found promising in earlier 650°F. tests[12] was selected for use in formulating a wide temperature range fluid.

APPROACH

Thermal Stability Tests

The thermal stability of the base fluid, tetra(2-ethylhexyl) orthosilicate and blends of additives in this fluid were evaluated in an Aminco high temperature shaking apparatus. The thermal stability test conditions were as follows:

1. Temperatures - 600, 650, 700 or 750 ± 10°F.
2. Duration - 6 hours.
3. Fluid volume - 30 ml.
4. Test cell - 180 ml. stainless steel.

5. Metal Catalysts - Copper, Steel, Titanium and Monel metal.

6. Atmosphere - Nitrogen of 99.997% purity.

After test, the fluid blends were compared on the basis of changes of the following:

1. Test system pressure during high temperature exposure.

2. Metal catalyst's weight.

3. Fluid viscosity at 100°F.

Additive Selection

An additive for enhancing resistance to hydrolysis and oxidation was essential for silicate fluids. Other desirable additives included those to improve the rubber swell, lubricity, and high temperature viscosity properties of the base fluid. Corrosion inhibitors were not considered since these types of chemica were expected to be thermally unstable at the projected test temperatures. Additives considered promising for improving base fluid properties were evaluated for their effect on the thermal stability of the base fluid and their ability to improve the base fluid properties in question.

Evaluation of a Finished Blend

A mixture that consisted of the base fluid plus useful, thermally stable additives, was then examined for other physical and chemical properties pertinent to hydraulic fluid use.

RESULTS

Thermal Stability Tests

1. Base Fluid - As shown in Table I, the tetra(2-ethylhexyl) orthosilicate was unstable at 750°F. Large viscosity losses and test system pressure increases due to volatile products of decomposition were noted. However, sufficient stability was evidenced at 700°F. to indicate this to be a feasible "target" test temperature for a silicate based blend. Experimental blends of promising additives in the silicate fluid were usually tested at this temperature.

TABLE I

THERMAL STABILITY TESTS[a]

Additive Blends in Tetra(2-Ethylhexyl) Orthosilicate

Additive and Conc., % by Wt.	Visc. Change @100°F,%	System Pressure Change, psig.[b] 1	2	3	Catalyst wt. Change, mg/cm.^{2} Cu	Ti	Steel	Monel
		RESULTS						
None[c]	-45	85	250	90	-.01	-.01	-.02	-.02
None	-23	30	135	35	-.01	-.01	-.03	0
Phenothiazine, 0.1	-17	25	145	40	+.02	+.02	0	+.03
Dioctyl-diphenyl amine, 0.1	-21	25	155	45	0	-.01	-.01	0
"Salicyl diamine," 0.1	-31	25	195	45	+.04	0	+.02	+.10
Phenyl α-naphthylamine, 0.1	-13	25	140	45	0	+.02	0	+.02
Phenyl B-naphthylamine, 0.1	-10	25	80	15	0	+.01	0	+.02
Phenyl B-naphthylamine, 0.2	-24	30	120	35	-	-	-	-
Phenyl B-naphthylamine, 0.5	-25	30	125	35	-	-	-	-
Phenyl B-naphthylamine, 1.0	-42	35	160	45	+.01	+.01	+.01	-.01
Decylol, 2.0	-55	30	190	50	-	-	-	-
Tetraphenyl silicate, 5.0	-39	20	180	50	-	-	-	-
Dicapryl phthalate, 10.0	-69	40	275	50	(Test ended after 4 hrs.)			
Mineral oil, 20.0	-44	40	185	55	-	-	-	-
Pyrene, 2.0	-13	35	155	45	-	-	-	-
Dibenzothiophene, 2.0	-23	30	170	45	+.01	0	+.02	+.03
Acenaphthene, 2.0	-14	25	125	35	-.01	0	0	0
Vinylpyrrolidone, 2.0	-30	25	190	55	0	+.02	-.01	-.02
Phenylsilicone-A, 30.0	-76	30	200	50	+.02	-.02	0	+.06
Phenylsilicone-B, 50.0	-74	35	140	40	0	+.01	+.01	+.03

(a) Test Conditions: 6 hours under N_2 at 700°F., except where noted.

(b) System Pressure: 1 = Initial at 700°F., 2 = after 6 hours at 700°F, 3 = Final after cooling to R.T.

(c) Test Temperature = 750°F.

2. Tests on Additive-Fluid Blends - Silicate fluids are subject to hydrolytic breakdown in the presence of water and have mediocre oxidation resistance. Several common antioxidants function as both antioxidants and hydrolysis inhibitors for silicates. Phenyl-α-naphthylamine (PANA) and dioctyl diphenylamine (DOPA) have been frequently used at concentrations ranging from 0.5 to 2.0%. These two, plus several others selected for this study, were evaluated in the silicate fluid at 0.1% by wt. concentration in a thermal stability test conducted at 700°F. Test results (Table I) indicated that the phenyl naphthylamines, PANA and PBNA (Beta-linkage), slightly enhanced base fluid stability. DOPA had little or no effect while the disalicylalpropylenediamine ("salicyldiamine") deleteriously affected the silicate's stability.

The PBNA was selected for further testing since it reduced formation of volatile products of decomposition, i.e., test system pressure rise was relatively low. This amine's effect on the thermal stability of the base fluid was tested at concentrations up to 1.0% by wt. (Table I). Test results indicated blend decomposition exceeded that of the base stock when PBNA concentration was raised from 0.5 to 1.0% by wt. since the viscosity and system pressure changes were considerably greater. The 0.5% concentration was therefore selected for the PBNA additive in the silicate fluid.

A second inherent problem in the use of silicate fluids is their inability to swell elastomeric gaskets and seals. It is desirable that fluids moderately swell such materials to more effectively seal the hydraulic system. Compounds were selected that are representative of types of chemicals known to increase rubber swelling and which were also likely to show adequate thermal stability. These included esters, mineral oils, alcohols and several aromatic compounds. Test results (Table I) indicated that an ester (dicapryl phthalate), alcohol (decyl), mineral oil (deep dewaxed, super refined) and an aromatic silicate (tetraphenyl) each acted to compromise base fluid stability when tested at concentration levels appropriate to each for securing significant rubber swell improvement. The pyrene, dibenzothiophene and acenaphthene appeared to have little effect on silicate stability.

A third problem related to the base fluid was its relatively low viscosity at high temperatures. Several polysiloxane fluids were thermally tested in the base fluid since they were considered promising for prospective use as thickeners. Test results (Table I) showed that the two phenyl methyl polysiloxanes (A-low phenyl content; B-high phenyl content) deleteriously affected the base fluid's stability.

In conclusion, the thermal stability tests indicated that the most compatible additives for the silicate fluid included the phenyl naphthylamines and chemically unreactive aromatic compounds.

Low Temperature Stability

It was desirable to use maximum amounts of aromatic additives in order to achieve maximum increase in the rubber swell properties of the base fluid. Silicate solutions of such chemicals that had been found to be thermally compatible were examined at several concentration levels. This was done to determine the additive's maximum solubility at low temperatures. The solutions were stored at a temperature of -90°F. for 72 hours and then visually examined for evidence of gel, precipitate or cloud formation. Such phenomena could not be tolerated since it would compromise the base fluid's superior low temperature fluidity. The maximum amounts of these aromatic chemicals that could be tolerated (were found to be completely soluble) at -90°F. were as follows:

Pyrene - 1% by wt.

Dibenzothiophene - 1% by wt.

Acenaphthene - 0.5 by wt.

The PBNA additive was also found to be soluble at 0.5% by wt. concentration at -90°F. Its upper limit of solubility was not established since previous thermal stability test results had precluded the use of this additive at higher concentrations. Quinalizarin (included as a metal passivator) was tested at 0.001% by wt. and found to be soluble at this concentration level.

Finished Blend Evaluation

Based on thermal and low temperature stability test results, a finished blend was evolved and identified as RIA 7M8 with the following formula:

Pyrene - 1% by wt.

Dibenzothiophene - 1% by wt.

Acenaphthene - 0.5% by wt.

PBNA - 0.5% by wt.

Quinalizarin - 0.001% by wt.

Tetra(2-Ethylhexyl) Orthosilicate - 97.0% by wt.

This blend was prepared by combining the components and heating with mild agitation to a temperature of 275-300°F. This blend was evaluated to establish its physical and chemical properties. Description of these tests and results follows:

1. Thermal Stability - The finished blend was tested under nitrogen (as previously described) at temperatures of 700°, 650° and 600° ± 10°F. Results are tabulated in Table II. The test data proved that at 700°F., the blend undergoes decomposition. However, at a temperature close to the recommended use temperature of 550°F., only slight deterioration of blend properties was found.

2. Rubber Swell - The blend was evaluated for rubber swelling by use of Federal Standards Method No. 3603.4. The volume increase of Standard Rubber L after exposure to the fluid for 168 hours at 158°F. was 12.6%. This value was nearly three-fold greater than that found for the silicate base fluid - 4.5%.

3. Hydrolytic Stability - The blend was examined by use of Federal Standards Method No. 3457. Results after exposure to water at 200°F. for 48 hours were as follows:

a. Cu strip catalyst, wt. loss, mg/cm^2 - 0.25

b. Fluid viscosity change, % - 1.6

c. Insoluble residue, % by wt. - 0.08

d. Neut. no. increase of fluid layer - 0.38

e. Neut. no. increase of water layer - 0.3

This data indicated that the blend possessed good stability in the presence of water. Property changes were well within the limits permitted for another silicate fluid, described by Specification MIL-H-8446B.(6)

4. Lubrication Properties - Antiwear properties of the blend were measured by use of ASTM Method No. D2266-64T. Load-carrying ability and EP Weld Point were evaluated by Federal Standards Method No. 6520. Mean wear-scar diameters for the blend, after tests on immersed 52-100 alloy bearings at 1200 rpm. for 1 hour at 167°F., were as follows:

TABLE II

THERMAL STABILITY TEST

Blend 7M8 After Six Hours Exposure

Temp., °F.	Neut No. Change mg KOH	Visc. Change, %	Thermal Effects: Test System Pressure, psig.(a) 1	2	3	Catalyst wt. Change, mg/cm.^{2} Cu	Ti	Steel	Monel
700	+0.70	-26.2	25	110	30	-	-	-	-
700	+0.9	-31.1	30	130	30	-.01	-.01	-0.02	+.01
700	+0.8	-21.0	25	90	25	0	+.01	+.01	+.01
650	-	- 8.7	15	40	10	-	-	-	-
600	+0.3	- 1.9	10	20	5	+0.01	+0.01	+0.01	+0.01

(a) System Pressure; 1 = Initial, 2 = After 6 hrs. at test temp., 3 = After cooling.

1 Kg. applied load - 0.34 mm.

5 Kg. applied load - 0.40 mm.

10 Kg. applied load - 0.67 mm.

20 Kg. applied load - 0.80 mm.

30 Kg. applied load - 0.87 mm.

40 Kg. applied load - 0.94 mm.

50 Kg. applied load - 1.09 mm.

The load-carrying ability in terms of the blend's MHL (Mean Hertz Load) was 17.6 Kgs. The EP-Weld Point was found to be 126 Kgs. These results indicated the fluid's lubrication properties were comparable to those of uninhibited mineral oils.

5. Corrosiveness and Oxidation Stability - Results (Table III) showed the blend to possess good oxidation resistance and excellent compatibility with the metal catalysts. Except for the neutralization number change, all property changes were well within the limits permitted for Specification MIL-H-5606B[3] fluids, which are tested at a lower temperature, i.e., 250°F.

6. Copper Corrosion - Results (Table III) showed the blend had no significant effects on copper.

7. Shear Stability - As expected, results (Table IV) showed the blend to be shear stable since no polymeric additives were present.

8. Flash and Fire Point - Results (Table IV) showed these properties to be superior to those of mineral oils of similar viscosity levels. For such mineral oils, flash points of 200 to 250°F. could be expected.

9. Spontaneous Ignition Temperature - Data (Table IV) from the literature[13] indicated this value for a fluid with a similar base stock to be considerably higher than a MIL-H-5606A fluid (703°F. as compared with 437°F.).

10. Evaporation Loss - Results (Table IV) showed the blend to have a desirably low vapor pressure since evaporation losses were low even at 300°F. Low temperature mineral oil based fluids such as those described by MIL-H-6083C[4] show evaporation losses approaching 70% when similarly tested at a lower temperature, i.e., 212°F.

TABLE III

CORROSIVENESS AND OXIDATION PROPERTIES OF 7M8 FLUID

Test	Test Method Ref.	Test Conditions	Results
1. Corrosiveness and Oxidation Stability of Light Oils (Metal Strip)	Fed. Test Method Std. No. 791a, Method 5308.5	Temp. °F = 350 Time; hrs = 72 Air flow rate; liters/hr. = 5 Metal Catalysts; Cu, steel, Al and Monel Metal	Viscosity change, % = +4.8 Wt. loss, % = 4.4 Insoluble residue, ml. = 0.1 Neut. No. increase, mg KOH = 0.25 Catalyst wt. change, mg/cm^2 Cu = +0.01 Steel = +0.01 Al = +0.02 Monel = +0.01
2. Copper Corrosion By Petroleum Products	ASTM Method No. D130-56	Time = 72 hrs. Temp. = 212°F.	Rating = 1b (slight tarnish)

TABLE IV

PHYSICAL STABILITY PROPERTIES OF 7M8 FLUID

Test	Test Method Ref.	Test Conditions and Results
1. Shear Stability	MIL-H-6083C, para. 3.5.6	Viscosity Loss, % = 0.4
2. Flash and Fire Point	ASTM Method No. D92-57	Flash Pt., °F, = 375 Fire Pt., °F, = 445
3. Spontaneous Ignition Temp.	Lit. Ref. (13)	S.I.T., °F, = 703
4. Evaporation Loss	ASTM Method No. D972-56	At 212°F, loss, %, = 1.9 At 300°F, loss, %, = 7.4
5. Foaming Characteristics	ASTM Method No. D892-63	At 75°F, ml. = 15 Collapse time, min. = 2 At 200°F, ml. = 5 Collapse time, min. = 1 At 75°F, ml. = 15 Collapse time, min. = 3
6. Cloud and Pour Point	ASTM Method No. D97-57	Pour Pt., °F. = $<$-90 Cloud Pt., °F. = $<$-90
7. Low Temp. Stability	Stored at -90°F. for 72 hours	Precipitate or gel formation, ml. = 0

11. Foaming Characteristics - Results (Table IV) showed the blend to have excellent resistance to foaming. Foam formation was within the limits permitted by most other specifications.

12. Cloud and Pour Point - Results (Table IV) showed the blend to have excellent low temperature properties since it flowed at -90°F.

13. Low Temperature Stability - Results (Table IV) showed the blend to have excellent low temperature stability since it remained fluid after 72 hrs. at -90°F.

14. Viscosity - Results (Table V) showed the blend's fluidity properties were excellent at very low temperatures but were mediocre at very high temperatures (the blend's viscosity was less than 1.0 at 450°F.).

15. Viscosity Index - Results (Table V) showed the blend's rate of change of viscosity with temperature to be better than most mineral oil fluids except those containing large amounts of polymeric V.I. improvers.

16. Density - Results (Table V) indicated that the blend's density property should be comparable to that of mineral oils.

17. Bulk Modulus - Data (Table V) from the literature[8] for another silicate fluid indicated the blend's compressibility properties should be comparable to those of mineral oils.

18. Coefficient of Thermal Expansion - Data (Table V) from the literature[8] for another silicate fluid indicated the blend's volume change due to heating should be comparable to that of mineral oils.

DISCUSSION OF RESULTS

The tetra(2-ethylhexyl) silicate base fluid was found to show an acceptable degree of thermal stability in 700°F. tests. However, this fluid shows only mediocre resistance to the action of water or oxygen, therefore stabilizing additives to enhance such resistance were essential. The thermal stability tests, herein reported, proved that several commonly used stabilizers may adversely affect silicate stability at 700°F. These adverse effects were also noted for other additives that included rubber swell and V.I. improver additives. Similar incompatibility had been observed in earlier 650°F. tests on a chemically related fluid,

TABLE V

PHYSICAL PROPERTIES OF 7M8 FLUID

Test	Test Method Ref.	Test Conditions and Results
1. Viscosity, Kinematic	ASTM Method No. D445-64	at -85°F, cs. = 6,465 at -65°F, cs. = 1,517 at -40°F, cs. = 346.9 at +100°F, cs. = 6.95 at +210°F, cs. = 2.28 at +450°F, cs. = 0.69
2. Viscosity Index	ASTM Method No. D567-53	154
3. Density	Pyncnometer	at 15.0C (59°F) gm/ml = 0.890 at 23.0C (73.4°F) gm/ml = 0.884 at 37.8C (100.0°F) gm/ml = 0.873
4. Bulk Modulus	Lit. Ref.(8)	253,000 psi.
5. Coefficient of Thermal Expansion	Lit. Ref.(8)	0.00048/°F.

hexa(2-ethylbutyl) disiloxane, used in some MIL-H-8446B fluids. Several such blends were evaluated at this Research Laboratory(10) and found to show lowered stability when they contained polysiloxane V.I. improver, and diester rubber-swell improver additives.

The experimental work revealed several unreactive aromatic chemicals to show the best thermal compatibility with the silicate. These were of interest since they acted as rubber-swell improvers. The amounts of such chemicals that could be used, was restricted to low concentration levels because of their limited low temperature solubility. For these reasons, a combination of several such aromatics were used at concentrations from 0.5 to 1.0% by weight. Thus, a measure of rubber-swell improvement was obtained from each one without creating a precipitation problem should the finished blend be subjected to prolonged, low temperature exposure.

V.I. improver additives such as the polyisobutylenes and polymethacrylates could not be used since they undergo thermal shear at temperatures below 450°F. A high temperature stable polyester was found to be insoluble in the silicate fluid. The polysiloxanes were both thermally stable and soluble in the silicate, however, they were found to affect the silicate's stability.

The lubricity tests indicated the finished silicate blend properties to be comparable to those of an uninhibited mineral oil. Exploratory antiwear tests were conducted using several common lubricity additives, i.e., tritolyl phosphate and a chlorinated biphenyl. These additives did not improve silicate antiwear properties at concentrations up to 1.0%. Higher concentrations were not evaluated since it was expected that such compounds would adversely affect the silicate's thermal stability. In fact, earlier tests[12] performed at 650°F. had indicated thermal incompatability with both of these additives. Furthermore, it was likely that most antiwear agents that act by forming an acidic precursor at the point of wear would tend to form acidic products upon thermal stressing and, in turn, would accelerate decomposition of the silicate base fluid.

The temperature range recommended for the experimental blend was selected on the following basis. The lower temperature was set at -75°F. since the fluid showed an extrapolated viscosity of 3000 centistokes at this temperature. However, the blend still flowed after prolonged exposure at -90°F. The upper limit of 550°F. was set for bulk oil temperature to allow a 150°F. safety factor when compared to the thermal tests at 700°F. This compensated for system "hot spots" which might be at a higher temperature than that of the bulk fluid.

The upper, recommended use temperature of 550°F. was also 50° lower than the 600°F. laboratory tests wherein only slight effects on fluid property changes were found.

Several of the experimental silicate blend's properties were superior to those of currently used, low temperature mineral oil based fluids. They included the following:

1. High temperature stability.
2. Flammability resistance.
3. Shear resistance.
4. Foaming resistance.
5. High temperature volatility.

In addition, physical properties such as density, bulk modulus, and thermal expansion were comparable to those of mineral oils, so that no special engineering design problems would be created by use of this fluid.

CONCLUSIONS

The effects of various chemical additives on the thermal stability and other properties of tetra(2-ethylhexyl) orthosilicate, were studied. Many of these additives adversely affected the silicate's stability. Several unreactive, aromatic compounds were found to improve the fluid's rubber swell properties and also to be thermally compatible with the fluid.

A finished blend, based on the silicate fluid, showed properties that were considered promising for use in hydraulic systems that operate over a wide temperature range.

RECOMMENDATIONS

Hydraulic fluids with properties comparable to those of the experimental blend No. 7M8 are recommended for introduction into Military channels.

APPENDIX

TEST METHODS AND REQUIREMENTS PROPOSED FOR THE WIDE-TEMPERATURE RANGE HYDRAULIC FLUID

A wide-temperature range fluid has been formulated which had desirable characteristics for hydraulic systems application. Therefore a list of test methods and property requirements was prepared to identify such a fluid. The requirements generally permit considerable deviation from the exact properties of the experimental fluid just described. However, they should adequately define a fluid that would have a similar potential for use in systems operating under conditions of wide temperature extremes.

This list will facilitate recognition and purchase of such fluids for introduction into Military channels in addition to being a basis for a Military specification, should these fluids become widely accepted.

Section I. - Application Recommendations

1. Temperature Range -

   (a) In closed systems, pressurized with nitrogen or helium, -75°F. to +550°F.

   (b) In open systems, -75°F. to +250°F.

2. Restrictions - These fluids should not be used where extreme pressure lubrication is required or in systems containing components fabricated of magnesium or lead.[14] For maximum use life, hydraulic systems should be cleaned and thoroughly dried before the fluid is installed. Desiccant type breathers are desirable on the reservoirs of "open" systems where this fluid may be used.

Section II. - Fluid Property Requirements and Tests

Methods	
1.1 Physical Requirements	

Property	Value	Test Method
Kinematic Viscosity at -65°F., cs	2000, max.	ASTM No. D445-64
Kinematic Viscosity at 100°F., cs	6.5 - 7.5	ASTM No. D445-64
Kinematic Viscosity at 210°F., cs	2.0 - 2.5	ASTM No. D445-64
Pour Pt., °F., max.	-90	ASTM No. D97-57
Flash Pt., °F., min.	350	ASTM No. D92-57
Fire Pt., °F., min.	400	ASTM No. D92-57

1.2 Physical and Chemical Requirements

1.2.1 Evaporation Loss. Using Method ASTM D972-56, at 300°F., the fluid loss shall be less than 10%.

1.2.2 Foaming. Using Method ASTM D892-63, the foam volume values shall be less than the following:

(a) Initial test at 75°F., volume, ml., max., 25

(b) Intermediate test at 200°F., foam volume, ml., max., 10

(c) Final test at 75°F., foam volume, ml., max., 25

Collapse times shall be less than the following:

(a) After initial test, min., 3

(b) After intermediate test, min., 2

(c) After final test, min., 4

1.2.3 Thermal Stability. The fluid shall be tested in a sealed system under the following conditions:

(a) Temperature, °F., 700 ± 10

(b) Time, hrs., 6

(c) Atmosphere, Nitrogen

(d) Metal Catalysts, Copper (QQ-C-576), Low Carbon Steel (QQ-S-636), Monel Metal (Ni-Cu Alloy), and Titanium (MIL-T-9046C Class 1).

Note: The monel metal catalyst can be used in the form of a wire to support the other three metals.

The changes in test system, fluid, and catalyst properties shall be less than the following:

(a) Test system pressure rise during test, psig., 100

(b) Fluid viscosity change, %, 40

(c) Fluid neut. no. change, mg. KOH, 1.5

(d) Metal catalyst wt. changes, mg/cm^2, 0.1

1.2.4 Corrosiveness and Oxidation Stability. Method Federal Standards No. 791a - 5308.5 shall be used with the following modifications:

(a) Temp., °F., 350°F.

(b) Time, hrs., 72

(c) Air flow rate, liters/hr., 5

(d) Metal Catalysts, Copper (QQ-C-576), Low-Carbon Steel (QQ-S-636), Monel Metal (Ni-Cu Alloy), and Aluminum (QQ-A-[illegible]55).

Note: The monel metal catalyst can be used in the form of a wire to support the other three metals.

The changes in fluid and catalyst properties shall be less than the following:

(a) Fluid wt. loss, %, 5.0%

(b) Fluid viscosity change, %, ±10%

(c) Fluid neut. no. change, mg KOH, 1.0

(d) Fluid insoluble residue, ml., 0.2

(e) Metal catalyst wt. changes., mg/cm^2, 0.1

1.2.5 Copper Corrosion. Using Method No. ASTM D130-56 and time and temperature conditions, respectively, of 72 hrs. and 212°F., the copper specimens shall show a rating of 1b or less.

1.2.6 Shear Stability. Using the method specified in Military Specification MIL-H-6083C, paragraph 3.5.6, the fluid viscosity loss shall be less than 1.0%.

1.2.7 Low Temperature Stability. The fluid shall be stored at a temperature of -90°F. and after 72 hours shall show no evidence of precipitate or gel formation and shall flow readily.

1.2.8 Swelling of Rubber. Using Method No. Federal Standards 791a - 3603.4 the % volume swell of Synthetic Rubber L shall be between 10.0 and 20.0.

1.2.9 Anti-Wear Properties. Using Method No. ASTM D2266-64T, the mean scar diameters shall be as follows:

Applied Load, Kgm.	Diameter, mm., max.
1	0.40
10	0.75
40	1.0

1.2.10 Particulate Contamination. Using the method specified in Military Specification MIL-H-6083C, paragraph 4.4.2.4, the number of particles shall be within the limits prescribed in Table V of that Specification.

LITERATURE CITED

1. Military Specification MIL-H-46004, "Hydraulic Fluid, Petroleum Base, Missile," Rock Island Arsenal, 25 April 1966, U. S. Army.

2. Military Specification MIL-H-81019B (ASG), "Hydraulic Fluid, Petroleum Base, Ultra-Low Temperature," Naval Air Engineering Center, 20 April 1966, U. S. Navy.

3. Military Specification MIL-H-5606B, "Hydraulic Fluid, Petroleum Base, Aircraft, Missile, and Ordnance," Naval Air Engineering Center, 1 July 1966, U. S. Navy.

4. Military Specification MIL-H-6083C, "Hydraulic Fluid, Petroleum Base, For Preservation and Testing," Rock Island Arsenal, 17 November 1967, U. S. Army.

5. Military Specification MIL-H-27601A (USAF), "Hydraulic Fluid, Petroleum Base, High Temperature, Flight Vehicle," Air Force Materials Laboratory, 7 January 1966, U. S. Air Force.

6. Military Specification MIL-H-8446B, "Hydraulic Fluid, Nonpetroleum Base, Aircraft," Wright Air Development Center, 16 July 1959, U. S. Air Force.

7. Military Specification MIL-L-14107B, "Lubricating Oil, Low Temperature, Weapons," Rock Island Arsenal, 20 July 1967, U. S. Army.

8. "Introduction to Hydraulic Fluids," Hatton, R., Reinhold Publishing Corporation, New York, 1962, pp. 214-225.

9. "Physical and Chemical Laboratory Evaluation of Silicate Base High Temperature Hydraulic Fluids," Baum, G., Wright Air Development Center, July 1957, WADC Report 55-89, p. 4.

10. "Thermal Stability of Eight High-Temperature Proprietary Fluids," LeMar, R., Rock Island Arsenal, 27 October 1964, U. S. Army, Rock Island Arsenal Report No. 64-3157, p. 10.

11. "Development of a Hydrolytically Stable High Temperature Hydraulic Fluid," Peeler, R., Kovacich, S., California Research Corporation, July 1957, WADC Report 57-119.

LITERATURE CITED

12. "Thermal Stability of Nine High Temperature Fluids and Their Intermixtures," LeMar, R., Rock Island Arsenal, January 1964, Rock Island Arsenal Report No. 64-231.

13. "Research on the Flammability Characteristics of Aircraft Hydraulic Fluids," Zabetakis, M., Lang, F., Imhof, A., WADC Report 57-171 Pt. III, March 1959.

14. "Effects of Metals on Lubricants," Christian, J., WADC, Report No. 54-576 Pt. II, February 1958, p. 11.

Unclassified

Security Classification

DOCUMENT CONTROL DATA - R & D

(Security classification of title, body of abstract and indexing annotation must be entered when the overall report is classified)

1. ORIGINATING ACTIVITY (Corporate author)
Rock Island Arsenal
Research & Engineering Division
Rock Island, Illinois 61201

2a. REPORT SECURITY CLASSIFICATION
Unclassified

2b. GROUP

3. REPORT TITLE
DEVELOPMENT OF A WIDE-TEMPERATURE RANGE HYDRAULIC FLUID (U)

4. DESCRIPTIVE NOTES (Type of report and inclusive dates)

5. AUTHOR(S) (First name, middle initial, last name)
LeMar, Ralph L.

6. REPORT DATE	7a. TOTAL NO. OF PAGES	7b. NO. OF REFS
September 1967	31	14

8a. CONTRACT OR GRANT NO.

b. PROJECT NO.
DA No. 1C024401A108

c.
AMS Code 5025.11.802

d.

9a. ORIGINATOR'S REPORT NUMBER(S)
RIA 67-2254

9b. OTHER REPORT NO(S) (Any other numbers that may be assigned this report)

10. DISTRIBUTION STATEMENT
This document has been approved for public release and sale; its distribution is unlimited.

11. SUPPLEMENTARY NOTES

12. SPONSORING MILITARY ACTIVITY
Rock Island Arsenal

13. ABSTRACT
Work was performed to formulate an extreme low temperature hydraulic fluid that would show better physical and chemical stability at high temperatures than do the commonly used low temperature mineral oil based fluids. A synthetic fluid, tetra(2-ethylhexyl) orthosilicate, was selected as a base stock for the fluid. Various chemicals were examined for their effects on the base fluid's thermal stability and other properties. These chemicals included oxidation inhibitors, hydrolysis inhibitors, thickeners, and rubber swell improvers. Thermal stability tests at 700°F. showed the silicate fluid to be adversely affected by several frequently used additives. Unreactive, aromatic compounds were found that were compatible with the silicate fluid and also improved its rubber swell properties. A promising blend, No. 7M8, was evolved and evaluated for properties pertinent to hydraulic fluid use. Results indicated that this blend's properties were generally as good as or better than those of standard low temperature mineral oil based fluids. An outline of requirements for a Military Specification was prepared. (U) (Author)

DD FORM 1 NOV 65 1473 REPLACES DD FORM 1473, 1 JAN 64, WHICH IS OBSOLETE FOR ARMY USE.

Unclassified

Security Classification

14 KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
1. Hydraulic Fluids						
2. Thermal Stability						
3. Silicate Fluids						
4. Low Temperature Fluids						
5. Additives						